

WHAT IS CLAIMED IS:

1. Apparatus for quantitatively and qualitatively enabling the analysis of a volatile substance encapsulated in a plurality of rupturable microcapsules each of which (a) has a rupturable polymeric wall; (b) has an outside diameter in the range of from about 0.01 microns to about 1000 microns and has a wall thickness in the range of from about 0.01 microns to about 100 microns; (c) contains from about 50% to about 97% by weight of volatile substance or solution of volatile substance; and (d) is releasably adhered to the surface of a semi-solid substrate section, comprising:
  - (i) a horizontally-situated reciprocatingly-movable horizontal substantially solid substantially planar surface located in the 'X-Y' plane associated with a driving means therefor for effecting a reciprocating motion of said substantially solid substantially planar surface at a controllable frequency  $\phi$  or set of frequencies,  $\phi_1, \phi_2, \phi_3, \phi_n$  (wherein n is an integer in the range of from 1 to about 20) for a determined period of time,  $\theta$ ;
  - (ii) substantially removably supported on said substantially solid substantially planar surface, said hollow enclosure means having a void space surrounded by a gas-impermeable horizontally-disposed base, a gas-impermeable horizontally-disposed lid and a gas-impermeable substantially cylindrical wall extending upwardly from and circumventing said base and extending downwardly from and circumventing said lid, said lid and/or said cylindrical wall having at least one exit port means and an entry port means therethrough, said hollow enclosure means being maintained in a stable, rigid, upright configuration during operation of said apparatus and being adapted to stably contain (I) a plurality of mobile solid-state spheres and/or ellipsoids each of which has a weight of from about 1 gm to about 100 gm, a density of from about 2 gm/cc to about 10 gm/cc, an average diameter of from about 0.5 cm to about 3.0 cm. and a surface hardness Knoop value in the range of from about 160 to about 220 and (II) inter-leaved between layers of said plurality of spheres and/or ellipsoids, semi-solid substrate sections having laminar surfaces, each of

which has adhered thereto a plurality of said volatile substance-containing rupturable microcapsules each of which has a surface hardness Knoop value in the range of from about 10 to about 20 and a microcapsule wall tensile strength several orders of magnitude less than the tensile strength of each of said solid-state spheres and/or ellipsoids, with the range of mass ratios of said plurality of spheres and/or ellipsoids:semi-solid substrate sections being in the range of from about 20:1 to about 100:1;

- (iii) analyte collection means located downstream from said hollow enclosure means and communicating with said exit port means thereof, consisting essentially of tube trapping means whereby analyte mixture components emitted from said hollow enclosure means during gas flow therethrough and simultaneous operation of said horizontally-situated oscillatably-movable horizontal substantially solid substantially planar surface are entrapped in said tube trapping means; and
- (iv) upstream from said hollow enclosure means or downstream from said analyte collection means, gas flow-effecting means for effecting the flow of gas sequentially (I) from a location upstream from said first entry port means; (II) through said first entry port means; (III) into said hollow enclosure means in a direction substantially perpendicular to the plane of said base; (IV) past each of said plurality of spheres and/or ellipsoids; (V) through said exit port means of said hollow enclosure means and (VI) into and through said analyte collection means.

2. A process for carrying out a collection of analyte for the purpose of effecting quantitative and qualitative analysis of a volatile analyte composition encapsulated in a plurality of rupturable microcapsules each of which (a) has a rupturable polymeric wall; (b) has an outside diameter in the range of from about 0.01 microns to about 1000 microns and has a wall thickness in the range of from about 0.01 microns to about 100 microns; (c) contains from about 50% to about 97% by weight of volatile substance or solution of volatile substance; and (d) is releasably adhered to the surface of a semi-solid substrate section, comprising the steps of:

- (i) providing the apparatus defined according to claim 1;
- (ii) placing into the void space of said hollow enclosure means (I) layers of a plurality of mobile solid-state spheres and/or ellipsoids each of which has a weight of from about 1 gm to about 100 gm, a density of from about 2 gm/cc to about 10 gm/cc, an average diameter of from about 0.5 cm to about 3.0 cm. and a surface hardness Knoop value of from about 160 to about 220 and (II) inter-leaved between layers of said plurality of spheres and/or ellipsoids, semi-solid substrate sections having laminar surfaces, each of which has adhered thereto a plurality of said volatile substance-containing rupturable microcapsules each of which has a surface hardness Knoop value of from about 10 to about 20 and a microcapsule wall tensile strength several orders of magnitude less than the tensile strength of each of said solid-state spheres and/or ellipsoids, with the range of mass ratios of said plurality of spheres and/or ellipsoids:semi-solid substrate sections being in the range of from about 20:1 to about 100:1;
- (iii) engaging said driving means for effecting a reciprocating motion of said substantially solid substantially planar surface at a controllable frequency  $\phi$  or set of frequencies,  $\phi_1, \phi_2, \phi_3, \phi_n$  (wherein n is an integer in the range of from 1 to about 20) for a determined period of time,  $\theta$ ;
- (iv) simultaneously with the engagement of said driving means for effecting an oscillating motion of said substantially solid substantially planar surface, upstream from said hollow enclosure means, or downstream from said analyte collection means, effecting the flow of carrier gas sequentially (I) from a location upstream from said first entry port means; (II) through said first entry port means; (III) into said hollow enclosure means in a direction substantially perpendicular to the plane of said base; (IV) past each of said plurality of spheres and/or ellipsoids; (V) through said exit port means of said hollow enclosure means and (VI) into and through said analyte collection means

whereby volatile substance components emitted from the microcapsules ruptured as a result of the spheres and/or ellipsoids abrading against them during operation of the apparatus are entrapped in said analyte collection means.

3. The apparatus of claim 1 wherein the gas flow effecting means is upstream from said hollow enclosure means by means of pressurizing the carrier gas upstream from said hollow enclosure means.
4. The apparatus of claim 1 wherein the gas flow effecting means is downstream from said analyte collection means using vacuum pump means located downstream from said analyte collection means.
5. The process of claim 2 wherein the flow of carrier gas is effected upstream from said hollow enclosure means by means of pressurizing the carrier gas upstream from said hollow enclosure means.
6. The process of claim 2 wherein the flow of carrier gas is effected downstream from said analyte collection means using vacuum pump means located downstream from said analyte collection means.
7. The process of claim 2 wherein said spheres or ellipsoids are spheres fabricated from stainless steel.
8. The process of claim 2 wherein the said volatile substance is a fragrance; the walls of said microcapsules are composed of acrylic acid-acrylamide co-polymers cross-linked with a melamine-formaldehyde—methyl ether pre-condensate, and said spheres or ellipsoids are spheres fabricated from stainless steel.
9. The process of claim 8 wherein each of the rupturable microcapsules has an average diameter of from about 2.0 to about 15 microns and a wall thickness of from about 0.2 to 2.0 microns.

10. The process of claim 7 wherein the semi-solid section is selected from a group consisting of a fabric section, hair follicles and simulated human epidermis section.
11. The process of claim 6 wherein the said volatile substance is a fragrance; the walls of said microcapsules are composed of acrylic acid-acrylamide co-polymers cross-linked with a melamine-formaldehyde—methyl ether pre-condensate, and said spheres or ellipsoids are spheres fabricated from stainless steel.
12. The process of claim 11 wherein each of the rupturable microcapsules has an average diameter of from about 2.0 to about 15 microns and a wall thickness of from about 0.2 to 2.0 microns.
13. The process of claim 12 wherein the semi-solid section is selected from the group consisting of a fabric section, hair follicles and simulated human epidermis section.
14. The process of claim 2 wherein the intensity of the function product in the headspace as a function of time is in accordance with the algorithm:

$$I = 10 \sum M_i \int \cot(2\pi\phi\theta) d\theta = 10 \sum M_i [\ln(\sin\{2\pi\phi\theta\})]$$

wherein  $M_i$  is the mass of an individual steel ball,  $\phi$  is the number of reciprocations per minute for the reciprocating shaker and  $\theta$  is the time elapsed from commencement of operation of the apparatus, in minutes.

15. The process of claim 2 wherein the rate of functional product entering the trapping means is in accordance with the algorithm:

$$\frac{dM}{d\theta} = 20\pi\phi M [\cot(2\pi\phi\theta)]$$

and the amount of functional product collected in the trapping means as a function of time is in accordance with the algorithm:

$$\ln M = 10 [\ln \{\sin (2\pi\phi\theta)\}]$$

wherein  $M$  is the mass of functional product collected in the trapping means,  $\phi$  is the number of reciprocations per minute for the reciprocating shaker and  $\theta$  is the time elapsed from commencement of operation of the apparatus, in minutes.

16. The process of claim 2 wherein the reciprocating motion of the substantially solid substantially planar surface is at a frequency,  $\phi$ , in the range of from about 200 to 300 reciprocations per minute, and the pre-determined period of time of operation of the apparatus,  $\theta$ , is in the range of from about 2 minutes to about 40 minutes.
17. The process of claim 20 wherein the reciprocating motion of the substantially solid substantially planar surface is at a frequency,  $\phi$ , in the range of from about 260 to 290 reciprocations per minute, and the pre-determined period of time of operation of the apparatus,  $\theta$ , is in the range of from about 4 minutes to about 30 minutes.
18. The process of claim 7 wherein at least one of the steel balls is coated with a metal passivation coating which inhibits stainless steel ball surface adsorption of functional product.
19. The process of claim 18 wherein the metal passivation coating comprises a silica.
20. The process of claim 18 wherein the metal passivation coating is a silica coating having a thickness in the range of from about 0.5 microns to about 2.0 microns.
21. The process of claim 18 wherein the metal passivation coating is a silicon coating having a thickness in the range of from about 120 to about 500 angstroms.

22. The process of claim 11 wherein the mass ratio range of mass of stainless steel spheres:mass of hair follicles is from about 35:1 to about 40:1.

23. The process of claim 10 wherein the mass ratio range of mass of stainless steel spheres:mass of fabric sections is from about 50:1 to 60:1.

24. The process of claim 13 wherein the microcapsules are coated with a cationic polymer.

25. The process of claim 14 wherein the microcapsules are coated with a cationic polymer.